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Filter Media for Collecting Diesel Particulate Matter

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FILTER MEDIA FOR COLLECTING DIESEL PARTICULATE MATTER

BY

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FORWARD

Particulate emissions from light duty diesel passenger cars and trucks are being regulated by the Federal Government. The emissions certification practice requires determination of gram per mile emission rates under simulated urban driving conditions. Analytical procedures must be defined which permit accurate, precise measurement of the total particulate emission rates. The Environmental Sciences Research Laboratory contributes to definition of these procedures through programs which

- examine the dynamics of particle formation and agglomeration in the exhaust of diesel engines, and

- determine the similarities and differences of mobile source particles in the ambient air and those collected in laboratory simulations of ambient conditions, and

- define and qualify procedures used to collect particulate matter from mobile sources and determine the importance of artifacts to the composition of collected particles.

This report examines seven commercially available filter media as applied for determining the mass emission rate of particulate matter from light-duty diesels in the certification practice. The media include examples of PTFE membrane filters, glass fiber filters, and PTFE coated glass fiber filters. Pressure drops, collection efficiences, adsorption characteristics, structural differences, and the mechanics of particle deposition are discussed.

A. H. Ellison

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ABSTRACT

On March 5, 1980, the U.S. Environmental Protection Agency published "Standard for Emission of Particulate Regulation for Diesel-Fueled Light-Duty Vehicles and Light-Duty Trucks" in the Federal Register. Analytical procedures for determinating of particulate emission rates were defined which include filtration of measured aliquots of the total air diluted exhaust stream. Criteria were established for acceptable filter collection based on comparison of weight gain of a primary filter to that of a downstream back-up filter for the measured sample volume. This study examined seven commercially available filter media as applied in diesel particulate emissions collection. The media included Millipore Fluoropore FA, Dexiglas, Gelman A-E, Ghia Zefluor lu, Pallflex T60A20, Pallflex TX40HI and Pallflex 400HO. The Fluoropore and Zefluor media are PTFE membrane filters and the others are glass fiber and PTFE coated glass fiber filters.

The filter media were examined by collecting particulate emissions from a light duty diesel passenger car driven over the urban dynamometer driving schedule specified for emission certification. Relative collection efficiencies were determined by comparing the weight gain of a common back-up filter (Fluoropore FA and Pallflex TX40HI) to the weight gain of each of the seven examined media as primary filters. A second series of tests were conducted to examine filter interaction with gas phase components by using constant primary filters (Fluoropore FA and Pallflex TX40HI) with each of the seven studied media as back-up filters. Further comparisons were made by examining the fraction of collected total particulate matter extractable with methylene chloride solvent.

Structural differences in the filters which influence particulate collection mechanisms, sample flow rates and pressure drops, were studied microscopically. Two media, the Pallflex T60A20 and Zefluor, were also examined microscopically with varying levels of particulate load to determine the effect of collected particles on the filtration of subsequent particles.

The results indicate that under the defined test conditions the membrane filters yield low gram per mile emission rates due to difficulties with collection of Federal Test Procedure phase 1 emissions, and the Gelman A-E glass fiber filter high rates due to adsorption of gas phase emissions. The mechanics of particle collection are similar for both the membrane and fiber filters as applied in this program. With the fiber filters, about 5 percent of the particulate matter was collected by diffusion deposition, 95 percent by direct interception. With the membrane filters, essentially all particulate matter was collected by direct interception.

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INTRODUCTION

Of the numerous procedures available for collecting particulate emissions from mobile sources (1), the one selected is generally dependent on the particle characteristic being examined. Filtration is often used for determining total particulate mass and chemical composition; centrifugation, impaction, and electrostatic precipitation are used for determining size distribution. Generally, the method for sampling motor vehicle particles involves dilution of the total exhaust flow with filtered ambient air prior to collection, permitting, to the maximum extent practical, particle-particle and particle-gas interactions similar to those which occur in actual road experience. Samples of motor vehicle particles collected from raw, undiluted exhaust, may be biased because both physical and chemical properties may be affected by temperature and dilution (2-7). Particle mass, chemical composition, and size distribution can be sensitive to dilution. The amount of organic matter in the particulate phase depends on temperature. High temperatures can cause evaporation/desorption or prevent condensation/adsorption of the organic compounds. Generally, temperature reduction and dilution occur simultaneously. If the temperature is reduced without dilution, attention must be given to losses resulting from thermal deposition on the heat exchange surfaces. Additionally, the condensed phase organics will maximize. Dilution also affects particle size because concentration and temperature influence the rate of agglomeration and of transformation from gas to solid/liquid phase.

On March 5, 1980, the U. S. Environmental Protection Agency published rules and regulations concerning the control of particulate emissions from light duty passenger cars and trucks (8). Included were procedures for sampling and analyzing emissions to determine the gram per mile (g/mi)* emission rate of total particulate matter from this category of motor vehicles. The recommended sampling apparatus is illustrated in Figure 1. Particulate emission rates are determined by filtration of a measured aliquot of air-diluted exhaust. Samples are collected during each of three phases of the Federal Test Procedure (FTP). Primary and back-up filters are used to establish acceptable levels of collection efficiency. If the back-up filter weight gain during testing is less than 5 percent of the total weight gain of the primary and back-up filters, only the primary weight need be used in the emission rate calculation, otherwise, the total weight gain of both the primary and back-up filters is used in the calculation.

This presentation examines various filter media only as they are used to determine total particulate emission rates in the light-duty diesel certification practice. Chemical transformations, for example, reaction of gaseous exhaust components with filtered particulate matter, are possible and,

*To conform to Federal Register practice, this paper will use volume units of cubic feet and distance units of miles rather than metric units.

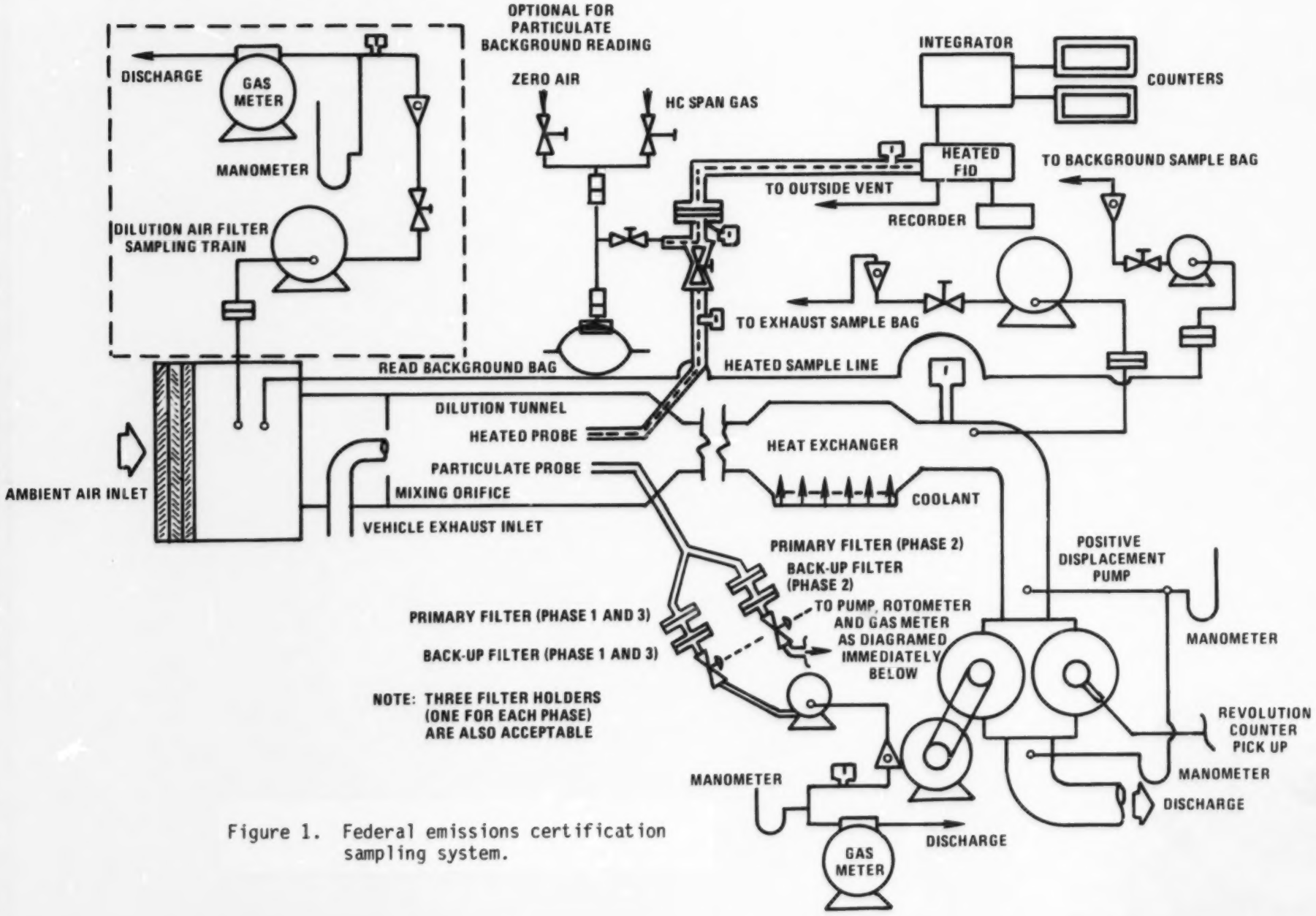

Figure 1. Federal emissions certification sampling system.

although their influence on the collected mass is not very large, they could be very important to the assessment of potential risk resulting from emissions to public health and welfare (2-4). Therefore, certification laboratory practices should be carefully examined to determine their suitability for collecting particulate matter for the biological assays used to estimate health risk.

Two primary filter types, membrane and fiber, are commonly used to collect diesel particulate from an exhaust stream. The mechanisms of particle collection are similar for both and have been studied for many years(9-12). Three major factors are important to the study of aerosol filtration: the dispersed particles, the dispersion medium (air for the case of interest), and the porous filtration medium. Important characteristics of the particles include their size or size distribution, shape, mass or density, electrical charge, chemical composition, and concentration. The gas flow is characterized by velocity, density, temperature, viscosity, and humidity; the porous filtration medium is characterized by surface area, thickness, size of structural units (e.g. thickness of fibers in fibrous filters), porosity, and electrical charge. Two major considerations in using filters for particulate collection are filtration efficiency and flow resistance or pressure drop. Both depend on the particle, gas flow, and filter characteristics previously mentioned.

Two mechanistic regimes exist when collecting particles by filtration: the first when deposition takes place on a pure, "unclogged" filter, and the second when deposition takes place on a "clogged" filter. The mechanisms for collection on "unclogged" filters include:

Diffusion deposition - collection through Brownian diffusion of the particle to the filter surface, not coinciding with the gas streamlines; the particles diffuse from the stream to the fibers and deposit thereon.

Direct interception - collection when a particle's center approaches a surface to within a distance equal to its radius.

Gravitational deposition - collection due to a sedimentary velocity resulting from gravity.

Electrostatic deposition - collection owing to the attractive forces of electrical charges.

Deposition by molecular forces - collection resulting from Van der Waals molecular forces; this is of primary importance in the adhesion of particles to surfaces.

Inertial deposition - collection due to particle inertia causing departure from the gas streamline and impaction on a filter surface.

The relative importance of these mechanisms depends on filter type, gas velocity, and the aerosol being collected. For example, with fiber filters, diffusion deposition is generally more important to the initial collection of the submicron particles emitted from motor vehicles than is direct interception.

Penetration of filters is usually discussed in terms of particle size, shape, and velocity. At constant velocity, as the particle size decreases in the submicron range, the degree of penetration increases to some maximum value, and then decreases with smaller particle sizes because diffusion increases. Similar peaks in penetration are observed as a function of velocity. With increasing velocity, penetration increases to a maximum value as diffusion becomes less important, and then decreases with the growing influence of inertia. An optimum sampling rate exists for any specific aerosol and filter medium. Particle shape can also be important. Spherical particles and long filamentous chain-like agglomerates, such as are common in diesel aerosols, behave differently in a gas stream. With chain-like agglomerates, deposition by diffusion or direct interception can depend on the orientation of the "filament" in the sample gas stream.

The mechanisms of collection change significantly when the filter becomes "clogged". Particles are deposited upon one another, dendrites form, and capillary effects or "grow-through" appear. This stage of collection is generally recognizable by increases in collection efficiency and pressure drop across the filter. By taking into account the vehicle particle emission rate (sample concentration), a sample flow can be selected to achieve filter "clogging" early in a test, thus attaining high average collection efficiency. The flow and resulting load must not be high enough to cause capillary penetration or grow-through losses. Care must also be taken to assure that the sample pumping apparatus can maintain constant flow for the duration of the sampling period. Filter "plugging" occurs when pumping capacity becomes inadequate to maintain flow.

In general, the mechanisms of collection are similar for fiber and membrane filters, but the relative importance of the various mechanisms differs with the filter type. The main difference between the filter types is that with membrane filters the gas flow is through pore-like passages in a thin membrane material, rather than through the deep filament network of fiber filters. With the membrane filters, direct interception is the primary mechanism of collection for diesel aerosols, even with short sampling periods and low filter loads. Diffusion deposition is on the walls of the pores rather than to the surface of fibers. Diffusion deposition is the primary mechanism of collection with fiber filters until the filter reaches a "clogged" state at which time direct interception becomes the dominate process.

During the study herein reported, samples of both membrane and fiber filters were examined as they are used for collecting particulate matter from diesel passenger cars in emissions certification. A representative single sampling rate was used throughout the study, so that all filters were examined with the same particle velocities. The particle dispersion medium was air with sample concentration and temperature varying as it normally does with the cyclic driving patterns and Constant Volume Sampling (CVS) systems required for emissions certification. Gravimetric procedures defined in the Federal Register were used throughout the study when determining filter weights (8). The pressure drop associated with each filter set (primary + back-up) was determined at the selected flow rate. FTP g/mi total particu-

late emission rates were determined with each filter type and differences examined by studying variations in apparent test phase total particulate and particle-bound organic concentrations. The relative collection efficiencies of the filters and the weight gains associated with adsorption of exhaust gaseous components were also studied. Vehicle driving conditions, temperatures, amounts of dilution, and sampling rates not included in the tests could all affect the observations. Final examination of the media included definition of important structural differences with a scanning electron microscope. Micrographs of front and back surfaces were prepared for each filter. Two filters, including a membrane and fiber filter, were examined with varying levels of diesel particulate load.

CONCLUSIONS

Seven filter media, two PTFE membrane filters, two PTFE coated glass fiber filters, and three standard glass fiber filters were tested for collecting diesel exhaust particulate matter using the procedures required for emissions certification. Particle composition, size distribution, concentration, and velocity at the filter face were held constant as were the dispersion medium composition, temperature and humidity, while the filter medium composition, thickness and porosity were varied.

Emission rates determined with the membrane filters were about 14 percent less than those determined with the fiber filters (excluding the A-E glass fiber value). The gas-tight seals in the filter holding devices may not have been adequate for the membrane filters because of their relatively thin structure, and this may have reduced their collection efficiency. However, the differences in the rates were so much greater during test phase 1 of the FTP than during the other tests phases that it suggests there may be differences in the interaction of the filters with the exhaust components. During test phase 1, the tailpipe particulate concentrations and temperatures, which affect agglomeration, size, shape and chemical composition, are different from those during tests phases 2 and 3. If the collection differences result from particle characteristics, the observations may change with varying sampling rates because of the sensitivity of the mechanics of particle deposition to particle velocity.

With the fiber filters, higher collection efficiencies were realized with the more dense, higher pressure drop filters. The Gelman A-E glass fiber filter appeared to gain weight through adsorption of gaseous components of the exhaust sample. The membrane filters appeared to give erroneously low g/mi particulate emission rates due to collection problems associated with test phase 1 of the FTP. The large standard deviations associated with the test phase 1 fiber filter results, however, would suggest further study is necessary before reasonable conclusions can be drawn. All of the examined media collected greater than 95 percent of the sample aerosol on the primary filter of a standard two filter collection set as determined by comparison of primary to primary plus secondary weights (Federal Register procedure).

Microscopic study of the media revealed significant structural differences. The glass fiber filters had different densities and fiber sizes, resulting in different pressure drops during sampling and slightly different collection efficiencies. Two of the examined glass fiber filters, Pallflex T60A20 and TX40HI, were PTFE coated. The TX40HI had significantly larger amounts of PTFE in the fiber system and also was backed with a PTFE coated

glass fiber "cloth" improving the filter strength. The PTFE membrane filters had average pore sizes about 1μ, nearly an order-of-magnitude smaller than the glass fiber filters. The membrane had a "quasi-fiber" structure with PTFE filaments about 0.2μ in diameter. The membrane filters had coarse backing materials so that the membrane itself could be as thin as possible to minimize sampling pressure drops, which still exceeded all of examined glass fiber filters. The Fluoropore filter used a polyethylene "ribbon" matrix for backing, while the Zefluor filter had a coarse PTFE "webbing". Microscopic examination also revealed the particle loads at which the gas flow passages become clogged, a condition important to the mechanics of deposition and collection efficiency. The fiber filter examined, T60A20, reached this condition at about .021 mg/cm^2, the Zefluor membrane filter at less than .004 mg/cm^2. Under the FTP test conditions used, more than 95 percent of particulate collection occurred under "clogged" filter conditions.

RECOMMENDATIONS

The two main filtration problems identified in the reported program were the collection of FTP test phase 1 (transient cold start) emissions with membrane filters and the adsorption of gaseous exhaust components with Gelman A-E glass fiber filters. Available resources would not permit comprehensive examination of either phenomenon. Further study is indicated, particularly of the collection of test phase 1 emissions. Comparison of test phase 1 data from the various filters showed scatter exceeding that of the other test phases. Further, several parameters important to deposition of particles on filters, for example particle velocity at the filter face, temperature and concentration, were not studied with the membrane or other media. Were the observations sensitive to particulate composition? Would similar results have been obtained with other motor vehicles and sampling conditions?

As indicated in this report, emphasis was placed on the accuracy with which total particulate mass emission rates can be determined with various filtration media. However, procedures defined for determining mass rates are commonly applied to collect samples of exhaust particulate matter for chemical analysis and assessment of potential health effects using various bioassay techniques. There are many possible physical and chemical phenomena associated with the laboratory sampling practice that could seriously bias results and estimates of the potential environmental impact of the emissions. Obviously, exhaust cannot be diluted in the laboratory exactly as it is on the roadway. The importance of dilution to mass has been examined and the laboratory procedures give reasonably accurate results. However, chemical changes occurring at low dilution are not well understood. Chemical kinetics would suggest that any process resulting in abnormally high concentrations of reactant compounds can affect composition. Further, it is possible that sampling system components, including the filter medium, can catalyze reactions. Substantial further study is indicated to determine the adequacy of certification procedures for collecting diesel particulate matter for determinations other than total mass.

EXPERIMENTAL RESULTS

Seven candidate filter media, listed in Table 1, were used to determine g/mi emission rates of total particulate matter from a four cylinder diesel passenger car using procedures provided for Federal certification.

TABLE 1. CANDIDATE FILTER MEDIA

Filter	Description
Millipore Fluoropore	PTFE membrane, 1μ
Ghia Zefluor	PTFE membrane, 1μ
Pallflex T60A20	PTFE glass fiber
Pallflex TX40HI	PTFE glass fiber
Pallflex 400H0	glass fiber
Gelman A-E	glass fiber
Dexiglas #225	glass fiber

The filter media included two polytetrafluoroethylene (PTFE) membrane filters and five glass fiber filters, two of which were PTFE coated. Both of the examined membrane filters had average pore sizes of 1μ.

Since the filter medium was the primary variable of the study, the other important parameters were held constant as much as possible. The particulate characteristics were controlled by conducting repetitive FTP emissions tests with a diesel passenger car using a standard CVS system as depicted in Figure 1. Although the particulate concentration and the dispersion medium temperature varied during the period of a test, which likely varied the time-dependent particle size distribution because they influence the degree of agglomeration, the test to test variability was minimal. The CVS flow rate and the driving cycle were kept constant.

Particle velocities at the face of the filter medium were maintained at 55.5 cm/sec. This corresponded to a 566.3 cm^3/sec (1.2 cubic feet per minute, CFM) sampling rate across a 10.2 cm^2 filter sampling area. This sampling rate resulted in final filter loads of 0.49 to 1.24 mg/cm^2, depending on the test phase and filter medium used. The pressure drop (without particles) associated with each filter set at this flow rate is reported in Table 2. Pallflex T60A20, being the least dense of the studied filters, had the lowest pressure drop at 24.5 mm Hg; the Zefluor membrane

TABLE 2. FILTER PRESSURE DROP

(combined primary & back-up filters)

Filter*	Flow rate, CFM	ΔP, mm Hg
Fluoropore	1.2	114.0
Zefluor	1.2	154.4
T60A20	1.2	24.5
Tx40HI	1.2	60.8
400H0	1.2	39.5
A-E	1.2	66.5
Dexiglas	1.2	52.5

*Used with Millipore 47mm filter holder, flow area 10.2 cm^2

filter had the highest pressure drop at 154.4 mm Hg. The pumping system utilized permitted maintenance of constant flow over this range of filter pressure drops for the duration of the sampling periods.

Table 3 presents the g/mi total particulate emission rates observed with each filter medium. The two membrane filters gave lower apparent emission rates than the five fiber filters (membrane mean value 0.425 g/mi, fibrous 0.514 g/mi). The difference in the mean values is statistically significant at the 99% confidence level. The g/mi emission rates were calculated using mean particulate concentrations of three or more repetitions on each FTP test phase (Table 4). The greatest difference between filter types occurred in test phase 1 (membrane average 0.63 mg/CF, fiber average 1.06 mg/CF), the

TABLE 3. FTP TOTAL PARTICULATE EMISSION RATES

Filter	Emission Rate, g/mi
Fluoropore	0.42
Zefluor	0.43
T60A20	0.52
TX40HI	0.49
400H0	0.51
A-E	0.56
Dexiglas	0.49

TABLE 4. FTP TEST PHASE TOTAL PARTICULATE CONCENTRATION

Filter	Test Phase Concentrations, mg/CF		
	I	II	III
Fluoropore	.632 ± .038	.314 ± .045	.513 ± .158
Zefluor	.625 ± .008	.323 ± .017	.559 ± .081
T60A20	1.05 ± .141	.326 ± .062	.619 ± .098
TX40HI	1.03 ± .41	.322 ± .066	.684 ± .131
400H0	.957 ± .485	.328 ± .055	.647 ± .063
A-E	1.25 ± .431	.321 ± .009	.688 ± .062
Dexiglas	1.02 ± .062	.288 ± .018	.700 ± .066
membrane average	.629	.319	.536
fiber average	1.06	.317	.669

cold start transient phase of the Urban Dynamometer Driving Schedule (UDDS) used for certification. Test phase 2 values were very close, and test phase 3 again gave somewhat lower apparent particulate concentrations with the membrane filters (0.54 mg/CF compared to 0.67 mg/CF). The Gelman A-E glass fiber filter gave the highest test phase 1 apparent concentration at 1.25 mg/CF. Test phase 1 standard deviations were significantly higher than either test phases 2 or 3, suggesting a possible deviation associated with the source and particulate characteristics rather than the sampling system and filter media.

One could hypothesize that when the vehicle is cold, larger amounts of organic material (lubricant, uncombusted fuel) are associated with the emissions and are collected to a greater extent by the fiber filters, possibly because Van der Waals molecular forces cause stronger adhesion of the particles to glass fiber surfaces. To test this hypothesis, test phase particulate matter was extracted with methylene chloride using previously reported Soxhlet extraction procedures (13). As indicated in Table 5, the apparent aerosol organic concentration was larger with the fiber filters in test phase 1, but it was not large enough to account for the differences observed in total particulate. During test phase 1, about 25 percent of the collected aerosol was organic with the fiber filters and 20 percent with the membrane filters; during test phase 3, about 25 percent was organic with both types of filters.

It is difficult to determine if the differences in amounts of organics result from differences in collection efficiency for the organic aerosol or from the greater adsorption of gas phase organics by some media than by

TABLE 5. FTP TEST PHASE PARTICULATE ORGANIC CONCENTRATION*

Filter	Test Phase Concentrations, mg/CF		
	I	II	III
Fluoropore	.126	.087	.126
Zefluor	.125	.083	.141
T60A20	.284	.087	.150
TX40HI	.220	.070	.165
400H0	.269	.104	.172
A-E	.262	.086	.178
Dexiglas		.084	.178
membrane average	.126	.085	.134
fiber average	.261	.086	.169

* $MeCl_2$ soluble particulate matter

others. Two sets of experiments were conducted to examine the filter relative particulate collection efficiencies and relative degrees of adsorption of "gaseous" components of the exhaust. One must interpret the following results with care since the procedures do not allow determination of when an organic droplet becomes small enough to be defined gaseous. In the first series of tests, repetitive hot start UDDS cycles were completed using each of the seven media in the primary filter position with a common back-up filter (see Figure 1). Although the back-up filter is not an absolute filter for the aerosol penetrating the primary filter, maintaining a common back-up filter should permit a reasonable measure of the relative collection efficiencies of the various primary media. Two back-up media were used, Fluoropore membrane filters and Pallflex TX40HI fiber filters. Results are reported in Table 6 for each phase of the hot start tests. The penetration is defined as the percentage of the total primary plus back-up filter particulate mass collected on the back-up filter. Generally, the fiber filters had higher relative collection efficiencies than the membrane filters, both as primary filters and as back-up filters for penetrant aerosol. The fiber filter efficiencies were ordered as were the ΔP values in Table 2; the more dense, higher ΔP filters had higher apparent collection efficiencies. The Gelman A-E glass fiber filter had the highest initial pressure drop and collection efficiency, the Pallflex T60A20 the lowest initial pressure drop and collection efficiency. Similarly, the Zefluor membrane filter had a higher initial pressure drop than the Fluoropore filter and a higher apparent collection efficiency. All filters indicated greater than 97 percent of the collected mass on the primary filter.

TABLE 6. RELATIVE FILTER PENETRATION*

Primary Filters	Back-up Filters: Fluoropore test phase+ I	Fluoropore test phase II	TX40HI test phase I	TX40HI test phase II	Average
Fluoropore	2.23	2.60	2.40	1.97	2.30
Zeflour	.78	1.18	1.44	1.43	1.21
T60A20	.83	1.12	1.44	1.61	1.25
TX40HI	.41	.26	1.38	0.99	.76
400H0	.69	.46	1.08	1.30	.88
A-E	.31	.45	1.25	.94	.74
Dexiglas	.63	.66	.97	.99	.81

* $\left[\frac{\text{back-up weight gain}}{\text{primary + back-up weight gain}}\right] 100$

+ Hot start UDDS cycles

A second set of experiments was conducted to examine the possibility of adsorption of gas phase exhaust components by the filter media. In these experiments the primary filter was held constant while the seven candidate media were used in turn as back-up filters. Again, it is difficult to determine if the observations are the result of collection efficiency differences for the penetrant aerosol or of gas phase adsorption. The data presented in Table 7 suggest a significant difference between the Gelman A-E glass fiber filter and the others examined, a difference owing to more than collection efficiency differences. The A-E glass fiber and the TX40HI PTFE coated glass fiber filter had similar relative collection efficiencies in previous tests, but substantially different weight gains in this series of experiments. The data suggest that adsorption of gas phase material contributes to the total mass measured with the A-E glass fiber filter.

The lower collection efficiencies observed with the membrane filters were not anticipated. Examination of these filters with monodispersed DOP aerosol by Liu, et al. suggests very high collection efficiency for particles of the size distribution of diesel exhaust aerosol (14). Differences in the mechanics of collection of spherical liquid droplets and long filamentous solid particles may account for the differences between anticipated and

TABLE 7. RELATIVE FILTER "ADSORPTION"*

Back-up Filters	Primary Filters: Fluoropore test phase I	Fluoropore test phase II	TX40HI test phase I	TX40HI test phase II	Average
Fluoropore	.31	1.91	.42	.84	.93
Zeflour	1.16	.55	.46	.06	.56
T60A20	2.15	1.47	.54	.94	1.28
TX40HI	1.71	1.97	.71	1.29	1.42
400H0	.61	.32	.29	.71	.48
A-E	5.74	5.00	4.96	4.32	5.01
Dexiglas	1.13	1.95	.76	1.73	1.39

* $\left[\frac{\text{back-up weight gain}}{\text{primary + back-up weight gain}}\right]$ 100

\+ Hot start UDDS cycles

observed findings. Since exhaust temperature and particulate concentrations are different in each test phase, agglomeration could vary the particle shapes in the different test phases. Possibly, test phase 1 particulate matter included longer chains, aligned with the gas flow, penetrating the thin membrane filters to a greater extent than the deep fiber filters. The thin structure of the membrane filter could also make a gas tight seal in the filter holder less certain. If all the measured sample flow were not through the filter medium, lower apparent collection efficiency would be indicated. However, as shown in Table 4, the membrane and fiber results compared very well in test phase 2, suggesting no significant contribution of sampling system leaks to the observed differences. If the differences resulted from particle size and shape, changes in parameters important to agglomeration could change the results, as would changes in particle velocities at the filter face.

Finally, the seven filter media were studied with an Amray 1000 Scanning Electron Microscope (SEM). Structural differences of the front and back surfaces of each filter were illustrated with micrographs of appropriate scale. Figures 2,3 and 4 illustrate the Pallflex 400H0, Dexiglas, and Gelman A-E glass fiber filters, respectively. As indicated in Table 2, the Pallflex T60A20 had the lowest pressure drop at a 1.2 CFM sample rate of this set, the Gelman A-E the highest pressure drop. Structurally, the three media had fibers of widely varying diameters. The back surface of the Pallflex 400H0 filter was noticeably different with well defined peaks and valleys resulting from processing to create a more uniform surface on a macroscale. Figures 5 and 6 illustrate the Pallflex T60A20 and TX40HI filters. The primary difference between these and other glass fiber filters is fluorocarbon coating of

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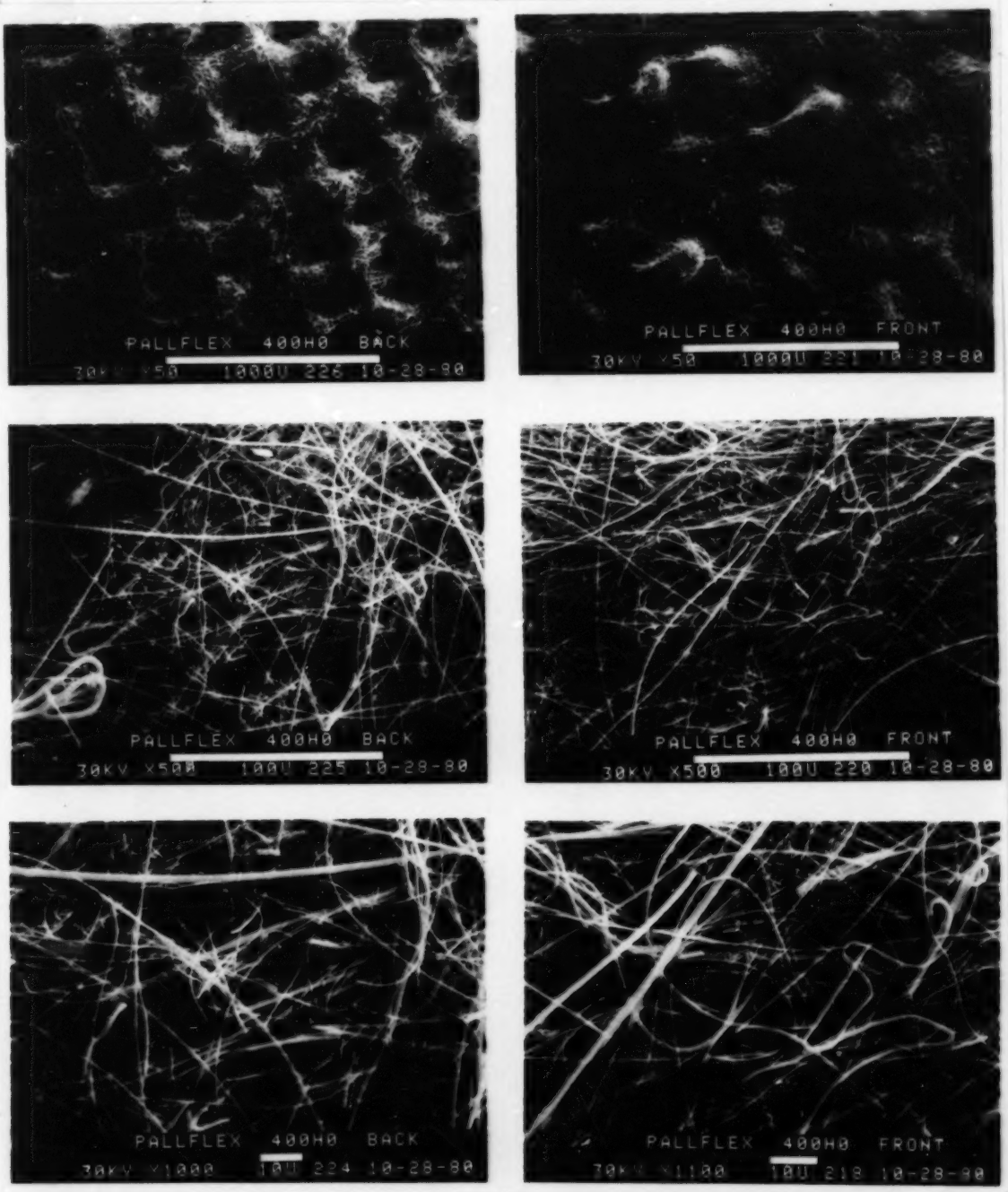

Figure 2. Pallflex 400H0 X50-X1000 micrographs.

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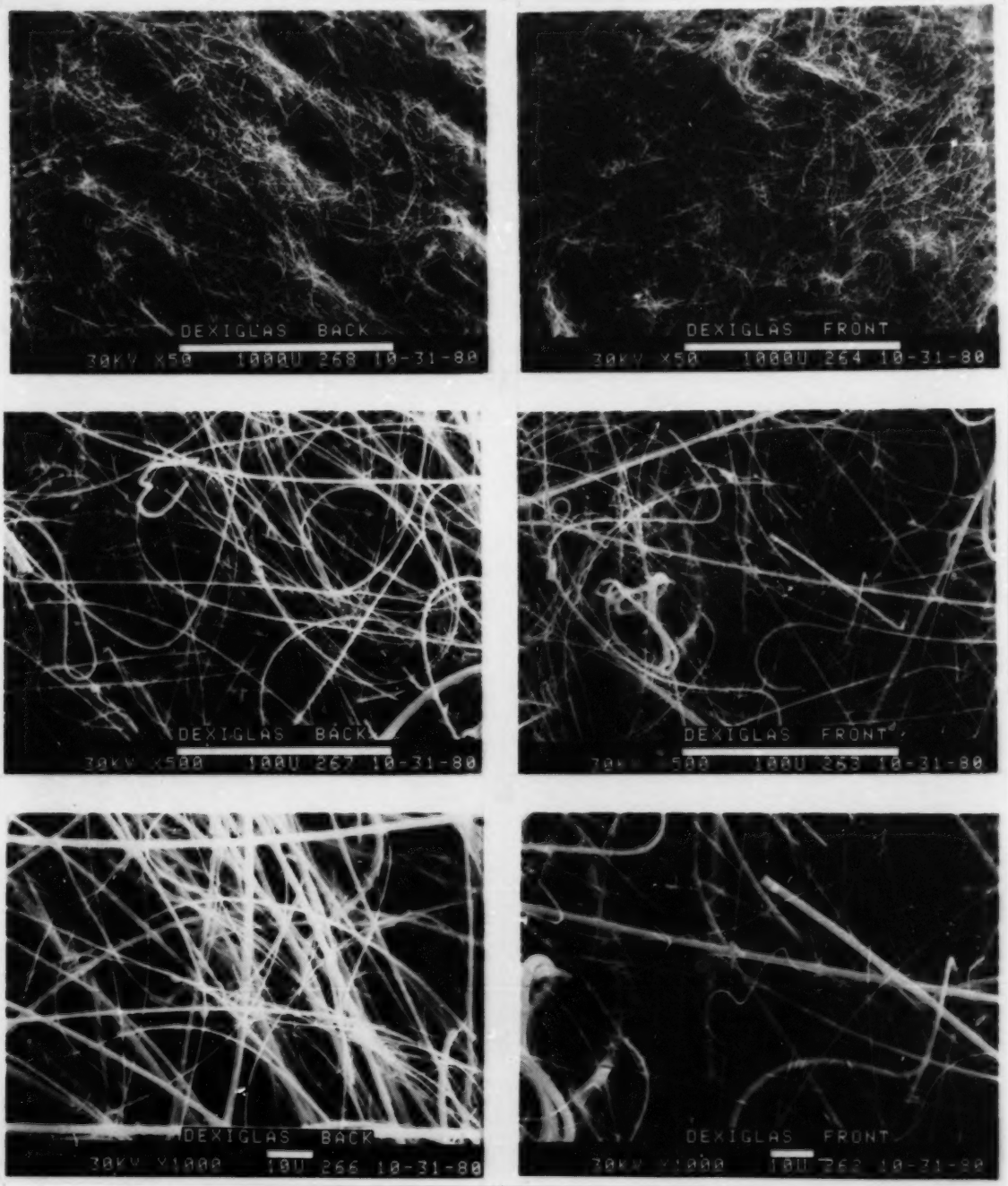

Figure 3. Dexiglas #225 X50-X1000 micrographs.

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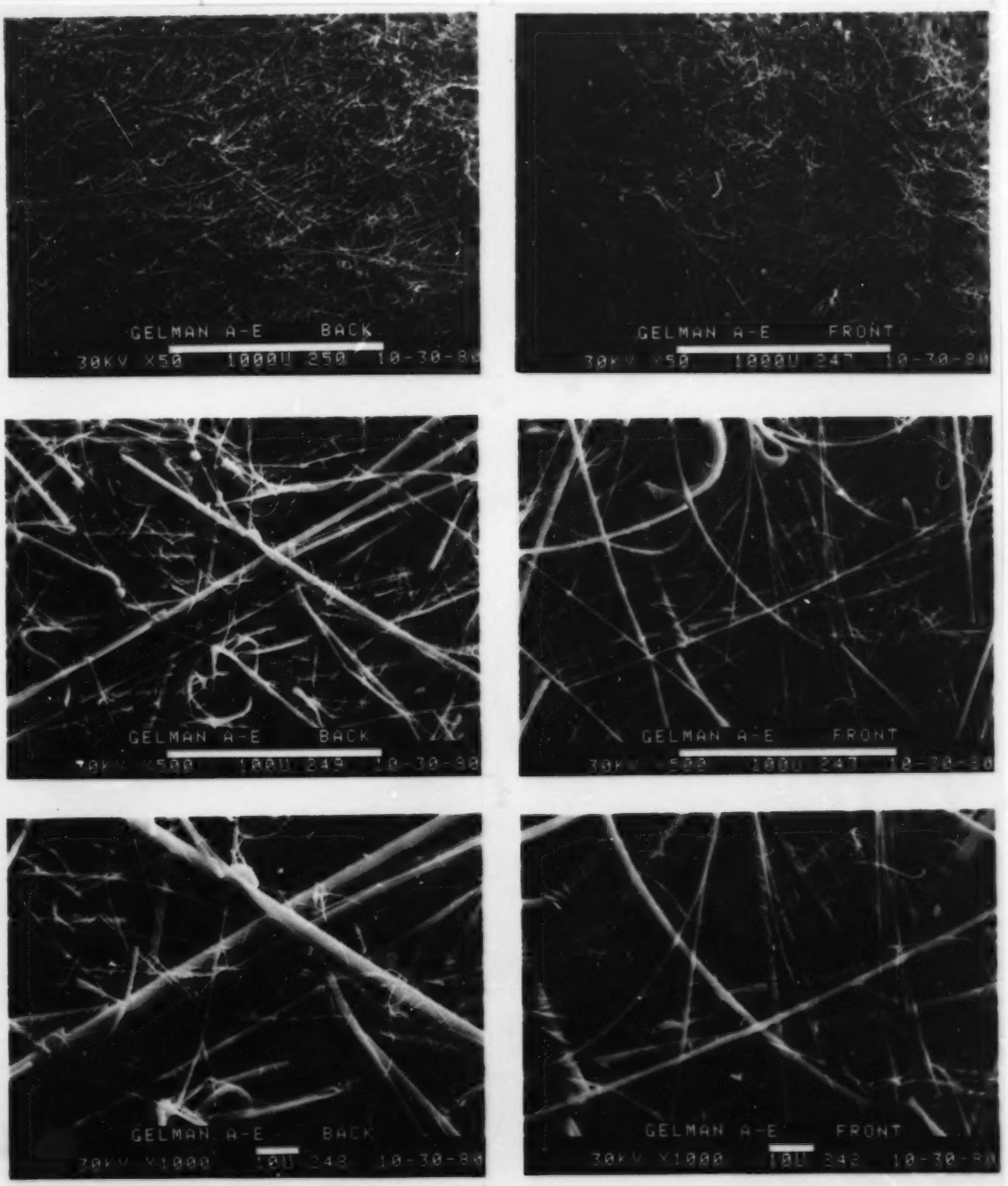

Figure 4. Gelman A-E X50-X1000 micrographs.

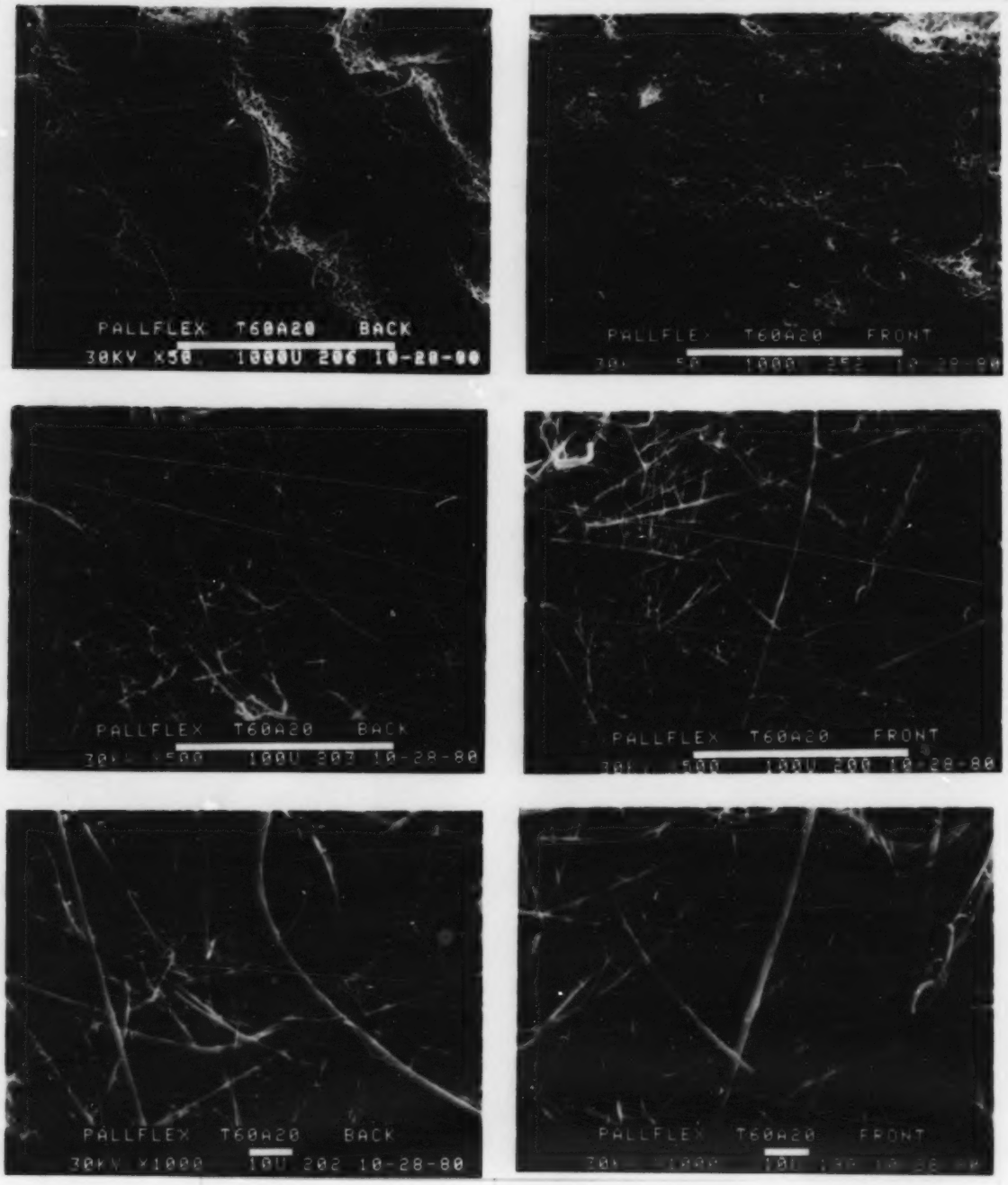

Figure 5. Pallflex T60A20 X50-X1000 micrographs.

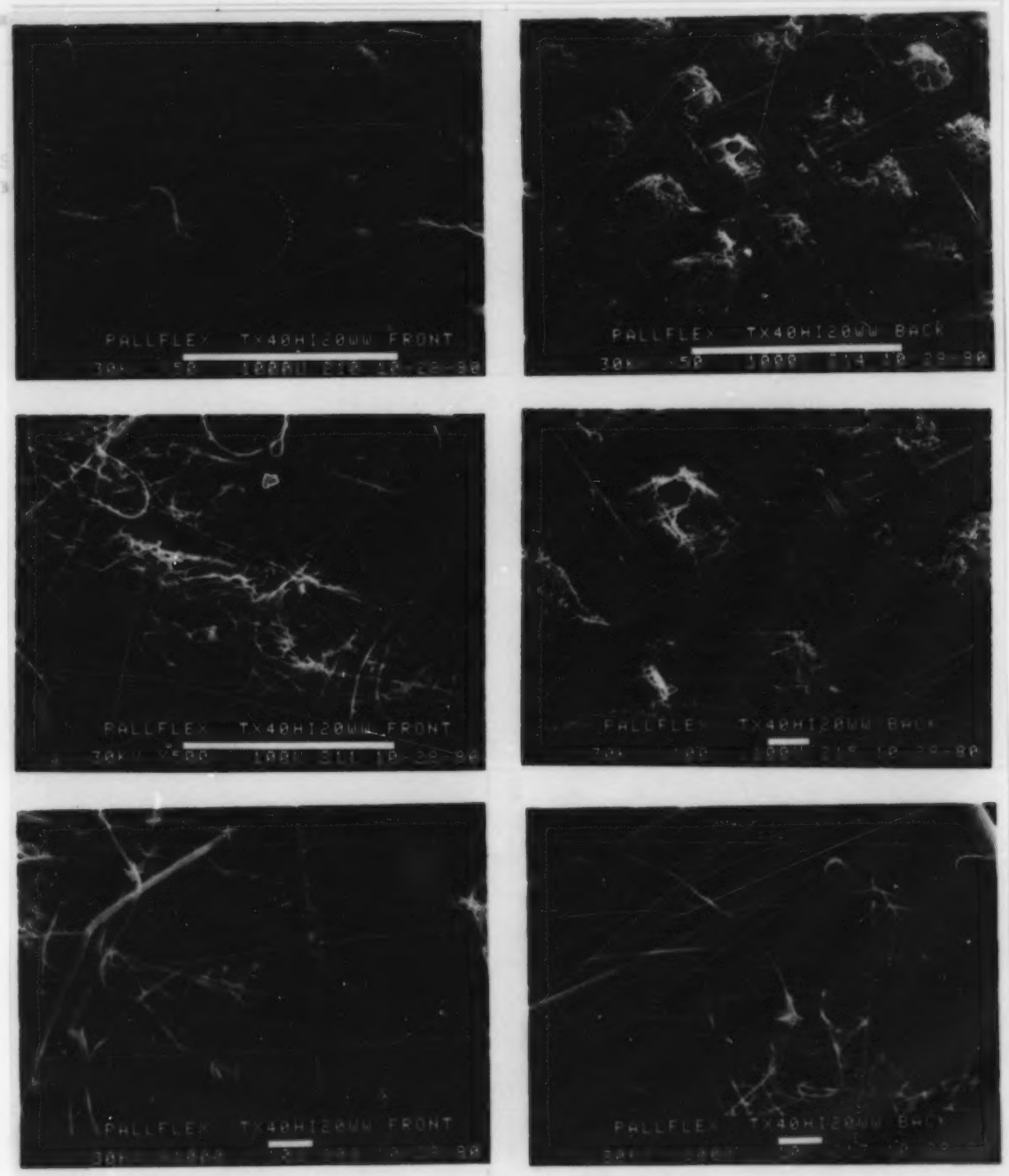

Figure 6. Pallflex TX40HI X50-X1000 micrographs.

the fiber surfaces. The PTFE coating binds the fibers rendering the filters stronger and more hydrophobic than uncoated glass fiber filters. The TX40HI filter has substantially more PTFE, readily apparent in the X500 and X1000 micrographs. Additionally, the TX40HI filter has a glass fiber "cloth" backing for added strength. The backing and increased PTFE results in greater pressure drops and collection efficiencies.

Figures 7 and 8 illustrate the Millipore Fluoropore FA and Ghia Zefluor PTFE membrane filters. Both require backing because the very thin primary membrane is extremely difficult to handle without some support, but the backings are different. The Fluoropore filter uses a polyethylene "ribbon" matrix, with "ribbons" about 100-200μ in width. The Zefluor filter is backed with a course PTFE webbing (note reverse labeling of micrographs). The average pore size of each filter is about 1μ. A striking contrast in structure appears when comparing the PTFE membrane X1000 micrographs with the glass fiber X1000 micrographs. The average "hole" in the membrane filter is at least an order-of-magnitude smaller than in the fiber filters, and the membrane filters are substantially thinner. An additional order-of-magnitude magnification of the membrane filters reveals a "quasi-fiber" structure with "fibers" about 0.2μ in diameter. The membrane "fibers" are relatively uniform in diameter, while the fiber filters have diameters ranging from less than 1μ to nearly 5μ. This type of membrane filter is commonly referred to as a tortuous pore membrane as opposed to a capillary pore membrane.

As previously discussed, fiber filters collect diesel aerosols mainly by diffusion deposition until the filter becomes "clogged" at which time direct interception dominates the collection process. A series of experiments were conducted to determine an approximate load at which filter "clogging" takes place with both fiber and membrane filters. The Pallflex T60A20 was used as representative of the fiber filters and the Ghia Zefluor as representative of the membrane filters examined. The pressure drop data indicate that the T60A20 filter was the least dense of the fiber filters examined and therefore would likely require higher particle loads to reach "clogging" than the others. To facilitate these observations the filters were loaded by sampling from a steady-state (45-mph) CVS diluted exhaust stream for varying lengths of time. The T60A20 and Zefluor samples were taken simultaneously with matched sampling rates. Table 8 presents the filter loads resulting from this test sequence. The loads were varied about from about .005 mg/cm^2 to about .05 mg/cm^2. The 15 second sample period gave filter loads somewhat higher than would be predicted by the 5, 10, and 60 second periods suggesting an elevated sample particulate concentration or sampling rate for the 15 second sample. The important parameter is filter load which was accurately determined. Figure 9 illustrates the T60A20 filter condition at .0050 mg/cm^2 and .0093 mg/cm^2. At the lowest load condition all of the particles are located on fiber surfaces with substantial surface area not loaded. At .0093 mg/cm^2 nearly all fiber surfaces are loaded and some of the fibers appear to hold multiple layers of particulate matter. The major gas flow "holes" are still open, however. In Figure 10 the filter appears to have attained a "clogged" condition at .0210 mg/cm^2. All of the "holes" are filled with particulate matter. Those areas of the filter which appear

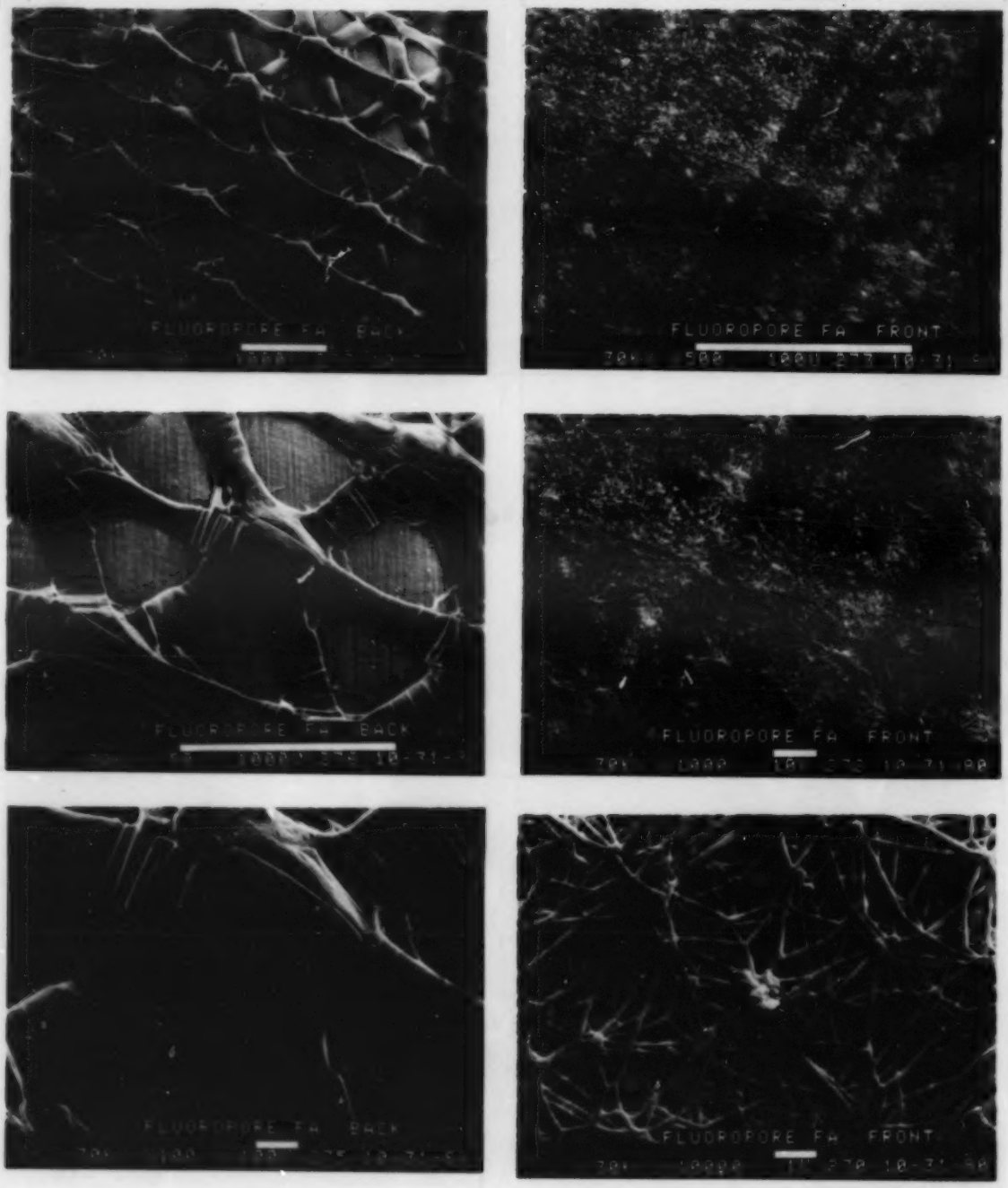

Figure 7. Millipore Fluoropore X20-X10000 micrographs.

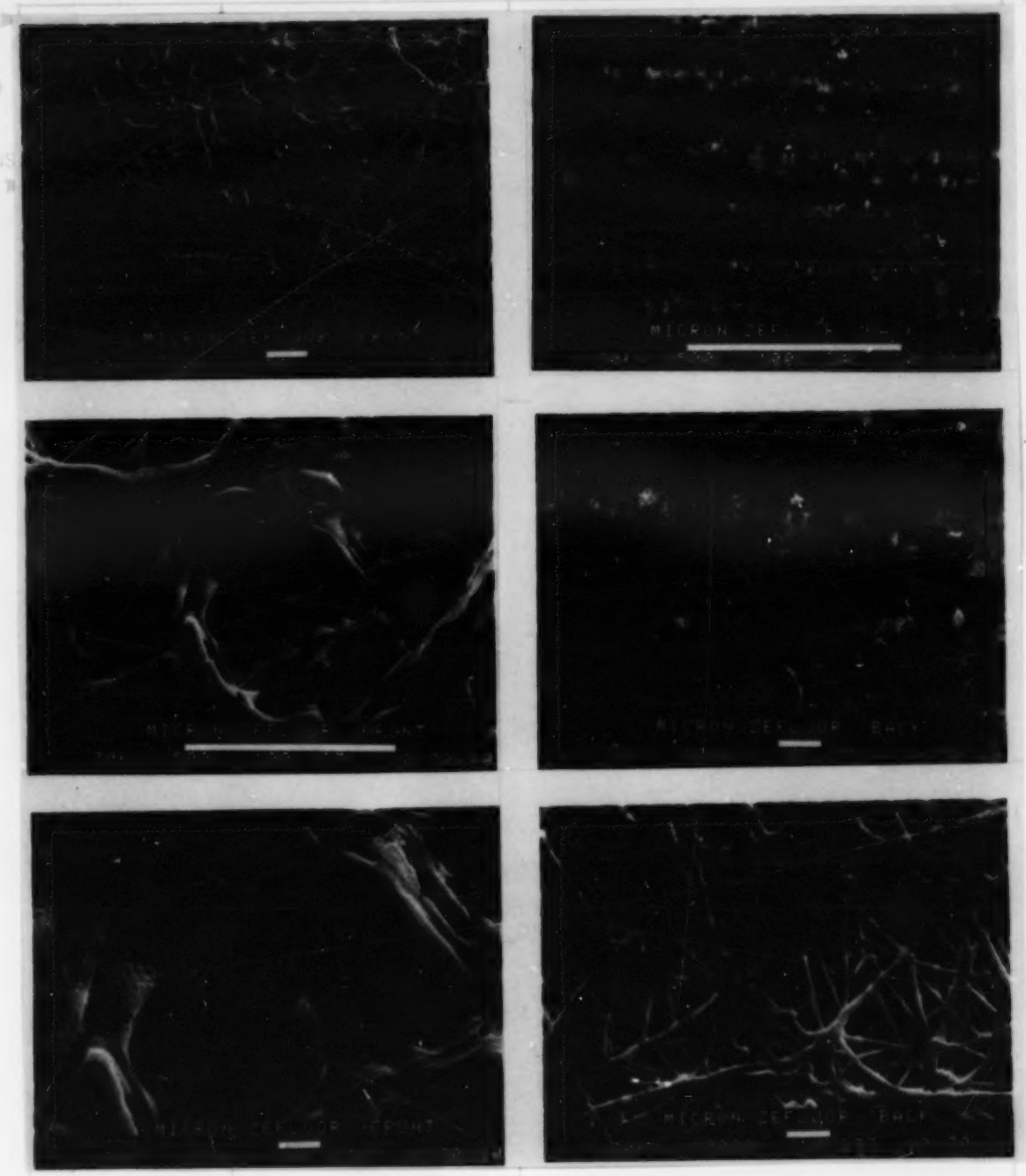

Figure 8. Ghia Zefluor X100-X10000 micrographs (front and back labels reversed).

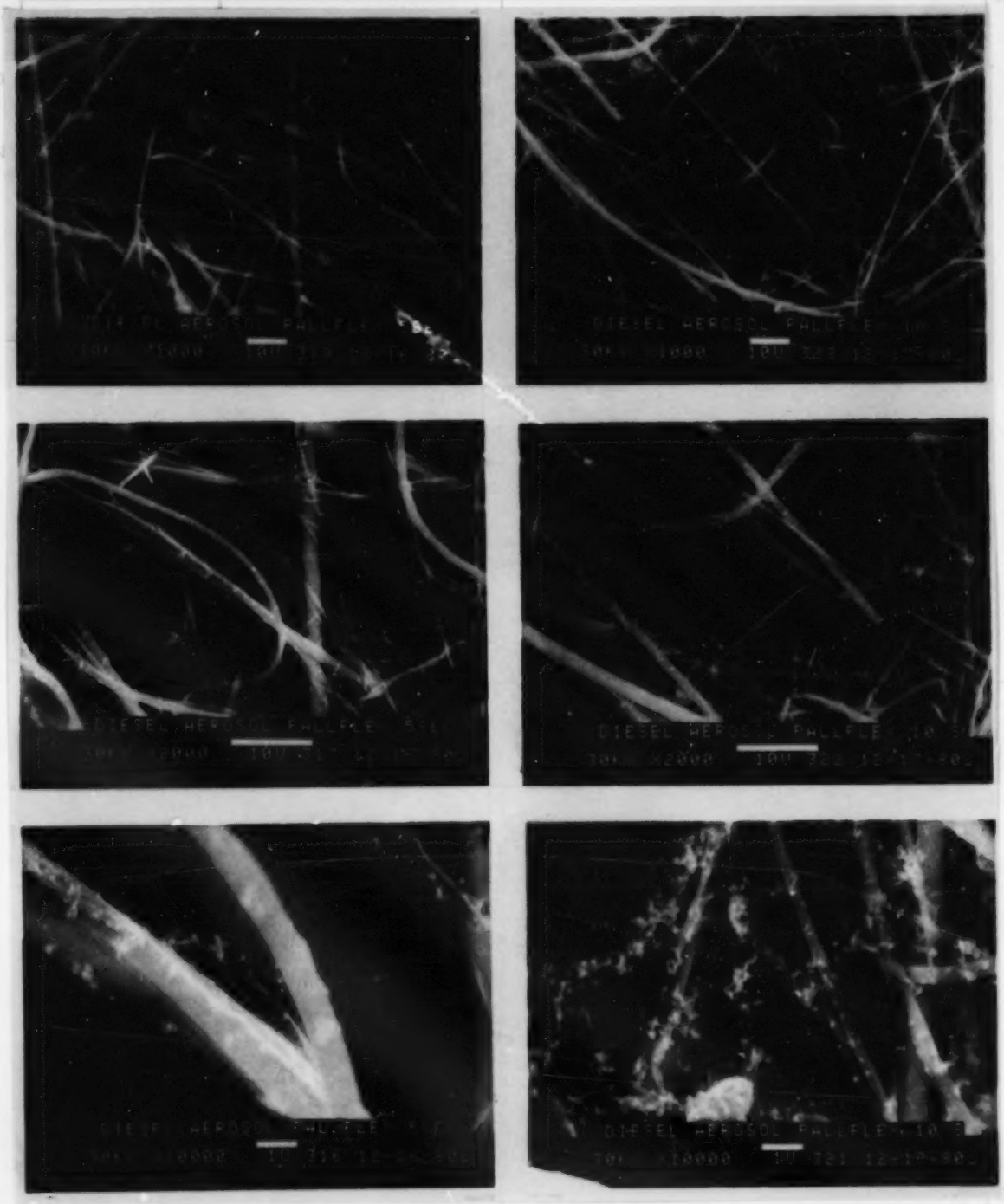

Figure 9. Pallflex T60A20 micrographs with 5 second and 10 second diesel particulate loads.

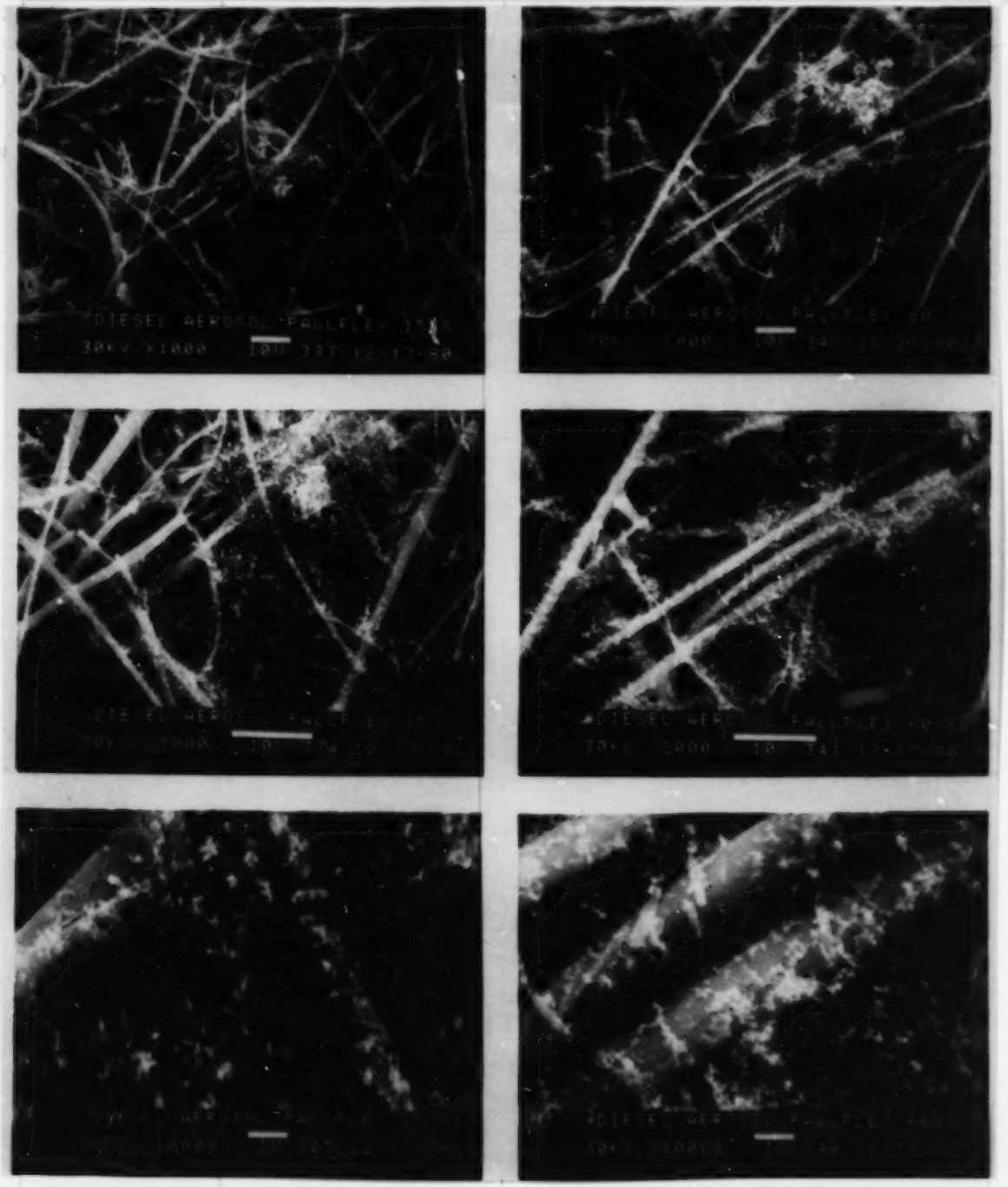

Figure 10. Pallflex T60A20 micrographs with 15 second and 60 second diesel particulate loads.

TABLE 8. FILTER LOADS FOR SEM STUDY, mg/cm^2

Sampling	Zefluor, 1μ	T60A20
5	.0040	.0050
10	.0081	.0093
15	.0200	.0210
60	.0509	.0531

free of particulate matter are actually closed to gas flow by PTFE. During the PTFE coating process, the film flow reached adjacent fibers, closing the "holes", and preventing gas flow during sampling. At this time the collection is primarily by direct interception, with particles collecting on particles. The Zefluor micrographs are not so interesting since even at the lowest load condition examined the particles are collecting upon one another. Figure 11 illustrates the 5 and 10 second samples along with a blank filter at the same magnification. At .0040 mg/cm^2 the filter surface is barely discernable, and at .0093 mg/cm^2 only particulate matter can be defined in the micrograph. Considering the final filter loads during vehicle emission rate testing, .49 to 1.24 mg/cm^2, at least 95 percent of the particulate collection occurred under "clogged" filter conditions.

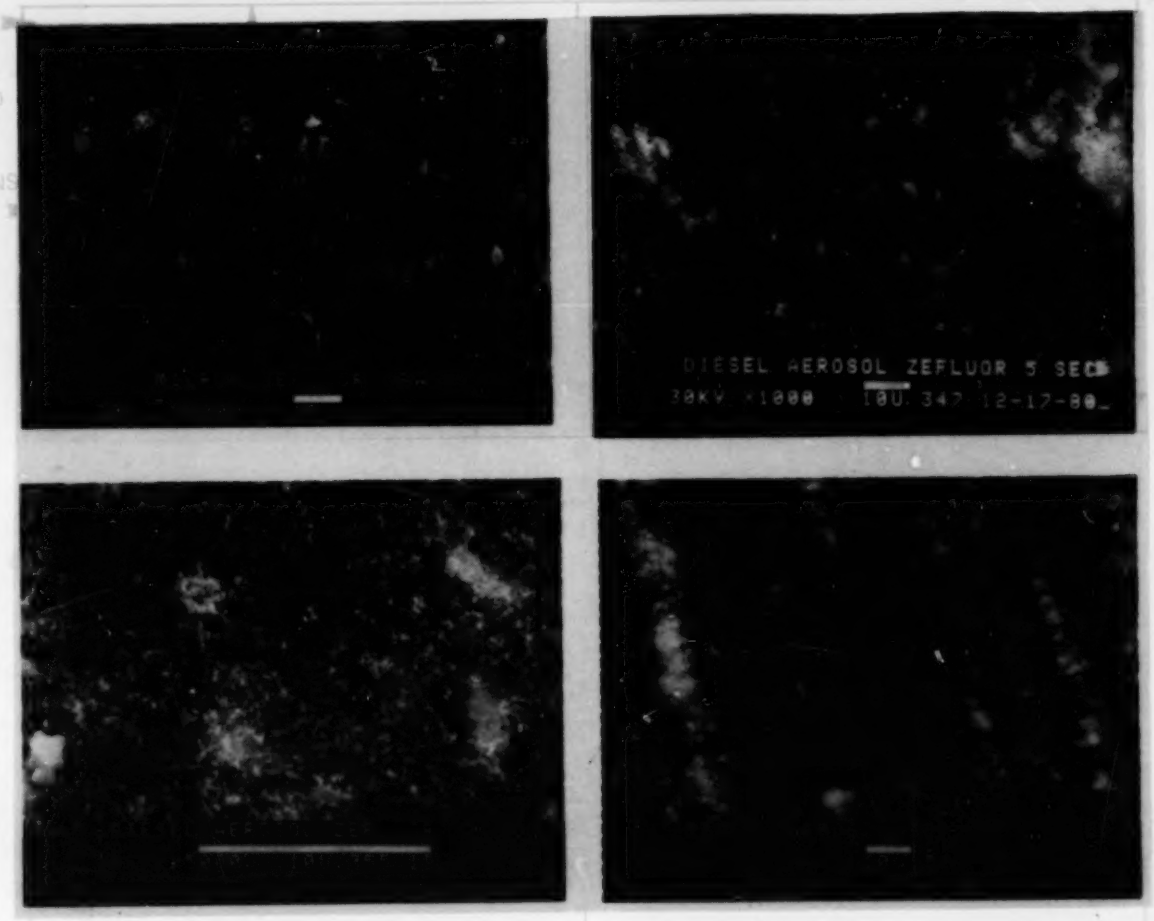

Figure 11. Ghia Zefluor micrographs with 5 second and 10 second diesel particulate loads.

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16. ABSTRACT

Certification of particulate emissions from diesel motor vehicles involves filtration of measured aliquots of the total air diluted exhaust. Seven commercially available filter media were examined for this purpose. The media included a variety of PTFE membrane filters, glass fiber filters, and PTFE coated glass fiber filters. Relative flow resistance (pressure drop), collection efficiency, and gas phase adsorption were examined. Filter structural differences, which influence particulate collection mechanisms, sample flow rates and pressure drops, were studied microscopically. Two media, a membrane and a fiber filter, were also examined microscopically with varying levels of particulate load to determine the role of collected particles on the filtration of subsequent particles.

The results obtained indicate that under the defined test conditions the membrane filters yield low gram per mile emissions rates due to difficulties with collection of Federal Test Procedure phase 1 emissions, and the Gelman A-E glass fiber filter high rates due to adsorption of gas phase emissions. The mechanics of particle collection are similar for both membrane and fiber filters as applied. Diffusion deposition is important with fiber filters for about 5 percent of the collected particulate matter, the remaining 95 percent collected primarily by direct interception. With the membrane filters direct interception is the dominate process.

17. KEY WORDS AND DOCUMENT ANALYSIS

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